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Technical Note
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Defence Research Establishment Atlantic

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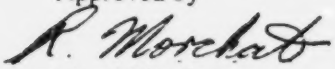

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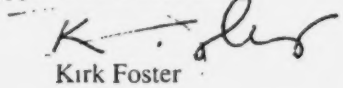

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Metallographic Study of the Plastic Zone Size Using Magnetic Etching and Visual Observation

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INTRODUCTION

To understand how fracture of ductile materials occurs, it is necessary to understand how the strain field, especially the plastic zone, develops around the crack as it advances by blunting and tearing [1]. This has been examined in a number of ways experimentally and by calculation. Lacking however, has been a technique, which is highly sensitive to plastic strain in metals, which can also be easily applied to a large area.

This paper describes the first stage of a program to overcome this difficulty. Briefly, it involves the magnetic etching of sections through cracked samples. Stainless steel AISI 304 is classified as an austenitic stainless steel grade because in its annealed state at room temperature its microstructure is entirely austenite. It is in fact a metastable austenitic stainless steel. Cooling to cryogenic temperatures or cold working can cause some transformation to α-martensite [2]. In contrast to the austenitic phase, the α-martensite is magnetic. This fact has allowed percent cold work on a macroscopic scale to be assessed non-destructively and on a microscopic scale, the local plastic strain to be estimated with confidence [3]. There are some important problems with this approach, notably that the transformation from which strain data is obtained is dependent on temperature [2], strain rate and composition. Provided all these are controlled, it should be possible to study the development of the plastic zone in fracture samples of annealed 304 stainless steel. Doing this requires the development and preparation of appropriate techniques for sample generation and the characterization of the samples.

This paper is divided into four sections. First, the materials composition and the sample preparation is outlined. Second, the precracking procedure and the known amount of

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deflection on each sample is given. Third, the results of the magnetic etching and visual observation are shown. Fourth, a final discussion is presented.

MATERIAL

The material used in this study was AISI 304 stainless steel. The composition limits [4] for this grade and the composition of the material used in these experiments are shown in Table 1. After the samples were machined (but prior to pre-fatiguing and the production of known amounts of blunting), the samples were heat treated to render them entirely austenitic. This was done by annealing at 1070 °C for 1 hour followed by quenching into iced brine. For this material a reasonable estimate of yield strength is 205.8 MPa (30 ksi).

Table 1. Chemical composition (%) of Stainless Steel 304

	C	Mn	Si	Cr	Ni	P	S
Specification Limits	0.08	2.00	1.00	18.0-20.0	8.0-10.5	0.045	0.03
Material Used	0.069	1.40	0.55	18.3	8.25	0.034	0

SAMPLE PREPARATION

Fatigue precracking is a method used to obtain reproducible sharp cracks in well defined plastic zones. In order to have a series of identical samples it is desirable to follow the same procedure as much as possible for each one. A fraction of the load P_L was used during the precracking. The frequency of loading was between 5 to 8 Hz. P_L is the load at which permanent bending will occur. For three point bend samples the following equation was used [5]:

$$P_L = \left[\left(\frac{4}{3}\right)\left(\frac{Bb_o^2\sigma_Y}{S}\right)\right]$$

Values of P_L were calculated for each crack length. From these calculated P_L values, appropriate applied loads for any crack length could be selected. Table 2 was used as a guide during the precracking of all the samples [6].

Table 2. Calculated values of P_L for different crack lengths.

	a		B		P_L		0.3 P_L	
	(mm)	(in)	(mm)	(in)	(kN)	(lb_f)	(kN)	(lb_f)
Start	2.4	0.094	23.0	0.91	18.3	4106	5.5	1232
	2.5	0.1	22.9	0.9	18.1	4050	5.4	1215
	3.8	0.15	21.6	0.85	16.1	3613	4.8	1084
	5.1	0.2	20.3	0.8	14.2	3200	4.3	960
	6.4	0.25	19.1	0.75	12.5	2813	3.8	844
	7.6	0.3	17.8	0.7	10.9	2450	3.3	735
	8.9	0.35	16.5	0.65	9.4	2113	2.8	634
	10.2	0.4	15.2	0.6	8.0	1800	2.4	540
	11.4	0.45	13.9	0.55	6.7	1513	2.0	454
	12.1	0.475	13.3	0.525	6.1	1378	1.8	413
End	12.7	0.5	12.7	0.5	5.6	1250	1.7	375

Once the precracking was successfully completed on the stainless steel 304 samples, each sample received a known load to cause crack to blunt. A variety of loads was used to give a range of blunting results. Sample number 1 was left in precracked condition to provide a reference point. Final blunting on each sample is given in Table 3.

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Table 3. Sample characteristics

Sample	Crack Length		Deflection		Final Load		Fraction P_L
	(mm)	(in)	(mm)	(in)	(kN)	(lb_f)	
1	11.4	0.45	0	0	1.8	400	0.3
2	11.4	0.45	0.23	0.009	8.5	1900	1.25
3	10.2	0.4	0.53	0.021	n/a	n/a	n/a
4	8.9	0.35	1.60	0.063	10.7	2400	1.14
5	7.6	0.30	6.0	0.24	23.5	5300	2.2

Once the 5 samples were precracked and load was applied, each sample bar was cut in half into two samples of half thickness. If more work had proven to be necessary half of the initial sample would be available for observation or experimentation. In order to eliminate further deformation to the samples, EDM wire cutter was used since this technique caused less distortion to the sample.

One half of each sample was then taken and reduced in size using The Powermet I abrasive cut wheel. The final sizing for the mount was made using a lower load system, the Isomet 200 precision saw. Again, the small abrasive wheel reduced deformation due to the cutting, preserving the experimental results.

Once the samples were mounted, they were cleaned and installed in groups of six on the automatic polisher, a Buehler Ecomet 3. Several methods were used but Table 4 shows the steps that were the most effective and produced the best results.

Table 4. Polishing procedure.

Polishing Material	Lubricant	Rotation
180 paper	Water	Contra
320 paper	Water	Contra
600 paper	Water	Contra
Texmet 2000	Metadi Supreme Diamond Suspension 9 Micron	Complimentary
Texmet 2000	Metadi Supreme Diamond Suspension 3 Micron	Complimentary

The samples were then polished using the Vibromet with Masterpolish for 8 hours

Two different techniques were used to reveal the plastic deformation around the crack tip so it could be reviewed in an optical metallograph. The first technique, magnetic etching, was based on the technique of R.J. Gray et al [7]. A drop of the water based with magnetized particles (60 Gauss) was applied to the polished surface of the sample and a glass cover slip was then installed on top. Micrographs were taken of the polished samples with the ferromagnetic colloid.

The samples were then electo-etched with a solution of oxalic acid (10g oxalic acid, 100ml distilled water). To further remove the effects of the initial polishing stages, the Vibromet and electro-etching steps were repeated, which greatly improved the results. Again, pictures were taken of each sample.

The final method used to compare the physical size of the plastic zone was using the recrystallization phenomenon [8]. Two half samples were selected from the five samples with the largest deformation. This ensured the best possible results. It has been confirmed in previous work [8] that 950 °C was sufficient for recrystallization and therefore the detection of plastic deformation. Samples #4 and #5 were placed in a

furnace at 950 °C for a period of two hours. The samples were then tap water quenched. Using the pre-established methodology, the samples were polished and electro-etched.

RESULTS

Sample number 1 was precracked but not loaded beyond the preckacking value of 30% of the yield load and therefore had no plastic deflection (δ) associated with it. It is clear from Figure 1 that Sample 1 was a very sharp crack, with a width of only 4 μm. On Figures 1a and b, the surface of the sample was electro-etched to reveal the microstructure. Even at a magnification of a thousand times, no grains were identified to have deformed. It is therefore very difficult to establish the plastic deformation radius (r_y) from the elctro-etched surface.

Figures 1c and d show the polished surface of Sample 1 with the ferro-fluid applied on the surface. When the coil was energized, the magnetic portion of the sample was revealed and measured to be r_y = 15 μm.

Sample number 2, displayed on Figure 2, had a permanent plastic deflection of δ = 0.23 mm. Figure 2a is a micrograph of the electro-etched surface at a magnification of 100 times. As with Sample 1, the size of the deformation radius r_y based on Figures 2a and 3a would be very difficult to determine. Figure 2b and 3b exposes the plastic deformation based on the ferro-magnetic particles and is evaluated to be 47 μm in radius. From the same figures it is calculated that the stretch zone width is approximately 28 μm in length. The stretch zone width is calculated by measuring the distance from the crack tip to the end of the yielded zone along the surface of the blunted crack.

Sample number 3 was deformed to a deflection of δ = 0.53mm. Again, from the electro-etched surface on Figure 4a, the geometry of the grains around the crack tip do not change enough to be able to conclude on the size of the plastic deformation. Figure 5 displays a

magnified view of the sample with the ferro-fluid on the surface, which helps to establish a plastic deformation size r_y of 85 μm and the stretch zone with of 50 μm.

Even with the high magnification micrograph (1000 times) from Figure 6a, the plastic zone would be hard to identify except for the slip lines easily identified on the right side of the sample. The slip lines identified on Figure 6a are confirmed with very dark zones of ferro-fluid on Figure 6. It is obvious when comparing Figures 6a and b that the plastic zone is much larger with the ferro-fluid.

Sample number 4 was deformed to a higher level than Samples 1, 2 or 3 with a permanent plastic deflection of 1.6 mm. The micrograph in Figure 7a is electro-etched and was magnified one hundred times. It is possible to evaluate a plastic zone based on grain elongation and slip lines with Figures 7a and b although the plastic deformation zone is bigger and more easily identified with ferro-fluid. The stretch zone width is evaluated to be 170 μm and the plastic zone radius, r_y, is 310 μm.

Sample number 5 sustained the most amount of deformation The deflection measured on the sample was $\delta = 6$ mm and to cause this amount of deformation, a load of 2.3 times the yield load value was applied to the sample. Massive deformation of the grains can be found on Figure 8a. Figure 8b shows the same amount of deformation and gives a plastic deformation size radius (r_y) of 1200 μm and a stretch zone width of 700 μm.

Portions of Sample number 5 are photographed at high magnification (1000 times) in Figures 9a and b The slip lines, which can be identified in Figure 9a, are easily identified and a similar micrograph using ferro-fluid (Figure 9b) by dark bands.

The third method employed to measure the physical size of the plastic zone was using the recrystallization phenomenon. Figures 10a and b display the results of the recrystallization. Due to the varying geometry of the grains in size and shape it is not possible to establish a clear boundary for the deformed plastic zones.

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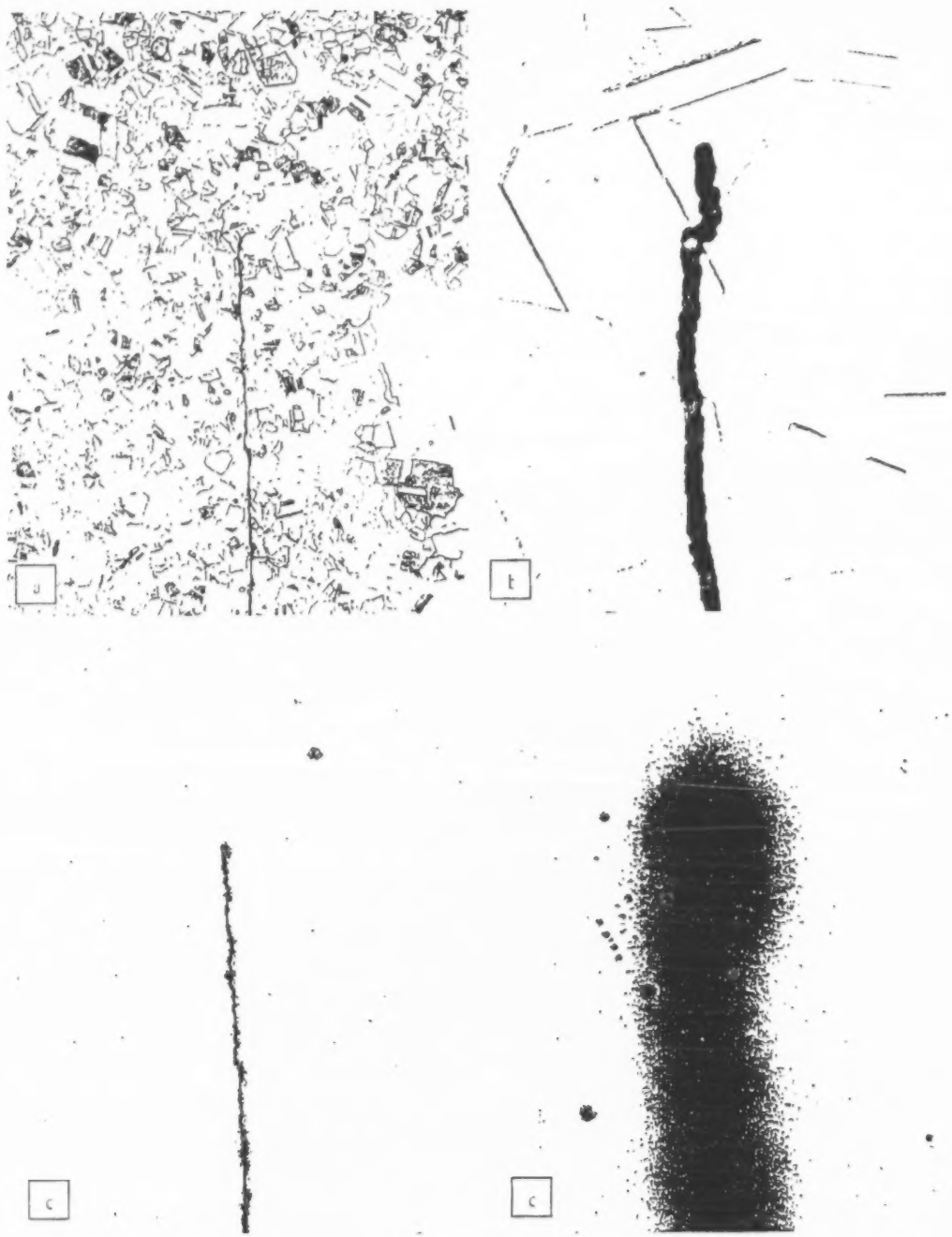

Figure 1. Sample #1, $\delta = 0$ mm, precracked reference sample (a. 100X electro-etched, b. 1000X electro-etched polished, c. 100X, ferro-fluid, d. 1000X, ferro-fluid).

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Figure 2. Sample #2, $\delta = 0.23$ mm, (a. 100X electro-etched, b. 100X, ferro-fluid).

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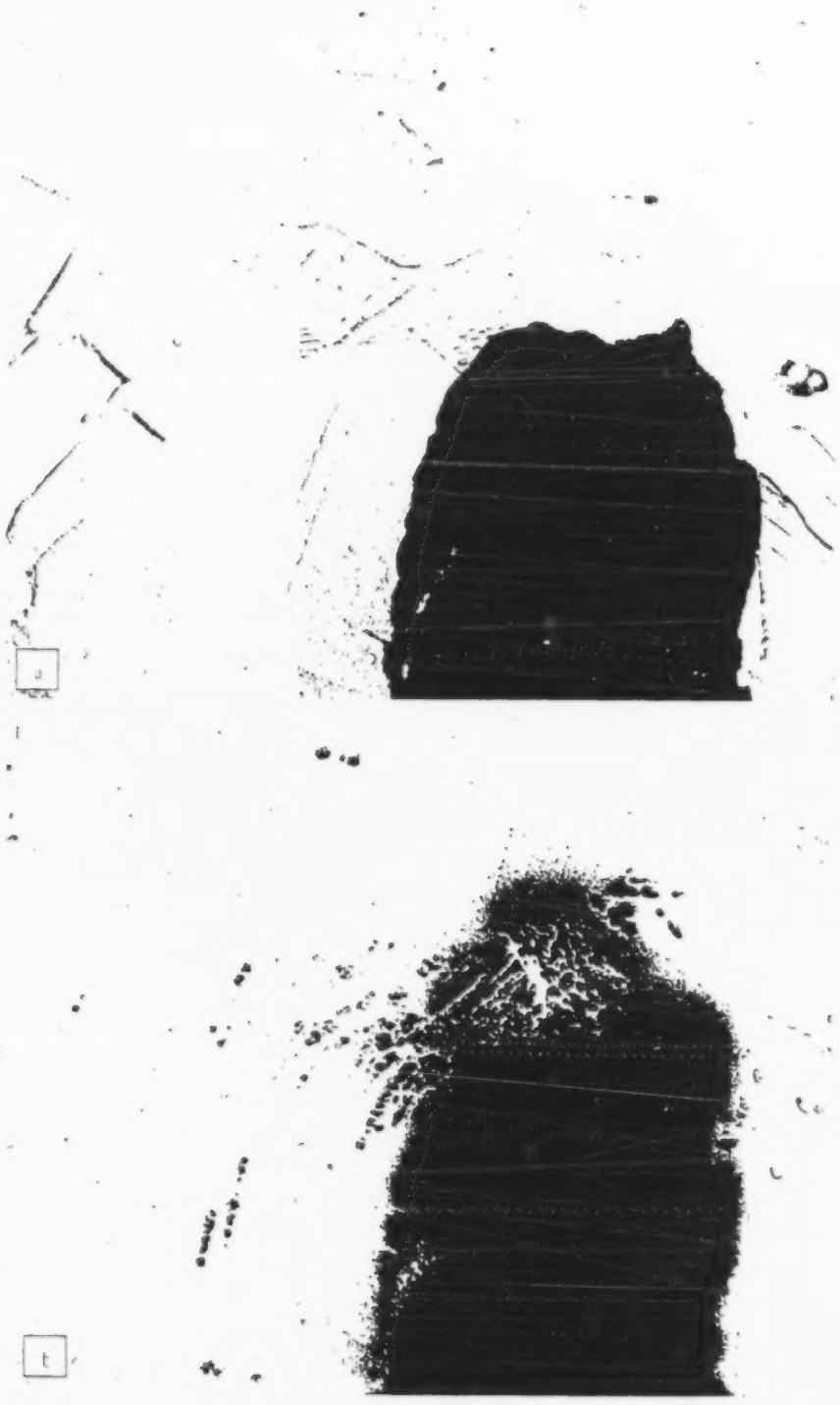

Figure 3. Sample #2, δ = 0.23 mm, (a. 1000X electro-etched, b 1000X, ferro-fluid).

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Figure 4 Sample #3, δ = 0 53 mm, (a. 100X electro-etched, b. 100X, ferro-fluid).

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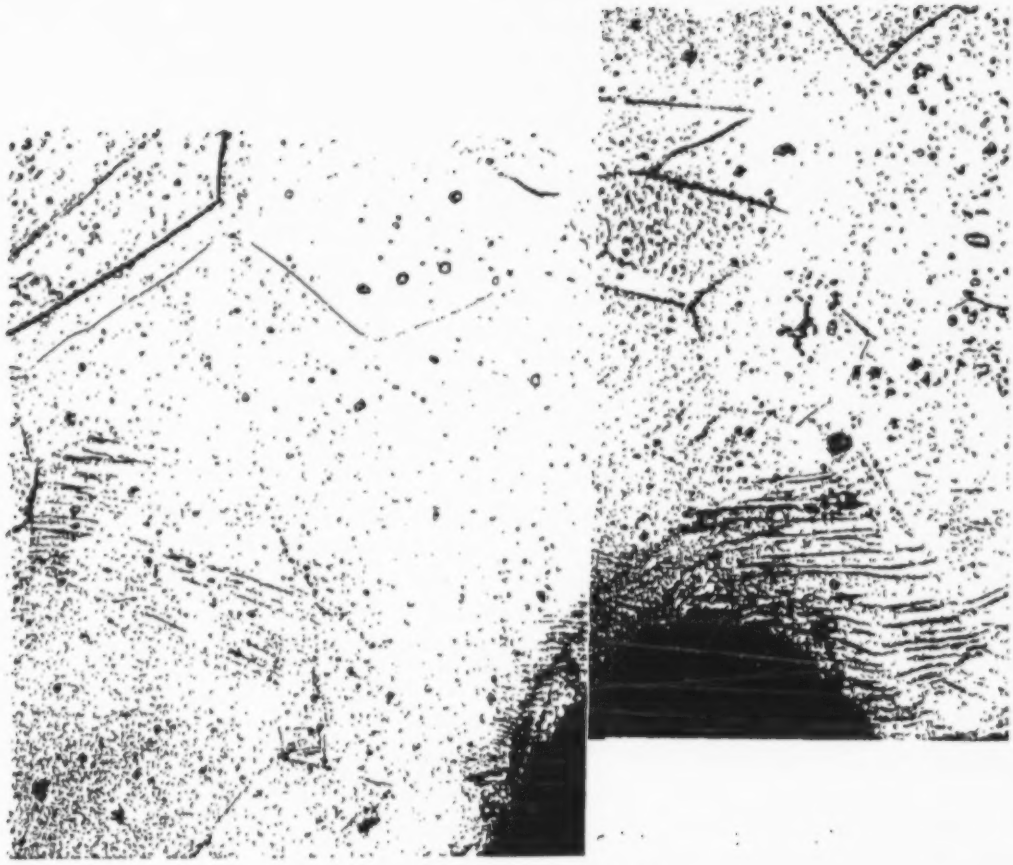

Figure 5. Sample #3, $\delta = 0.53$ mm, 1000X electro-etched.

Figure 6. Sample #3, $\delta = 0.53$ mm, 1000X ferro-fluid

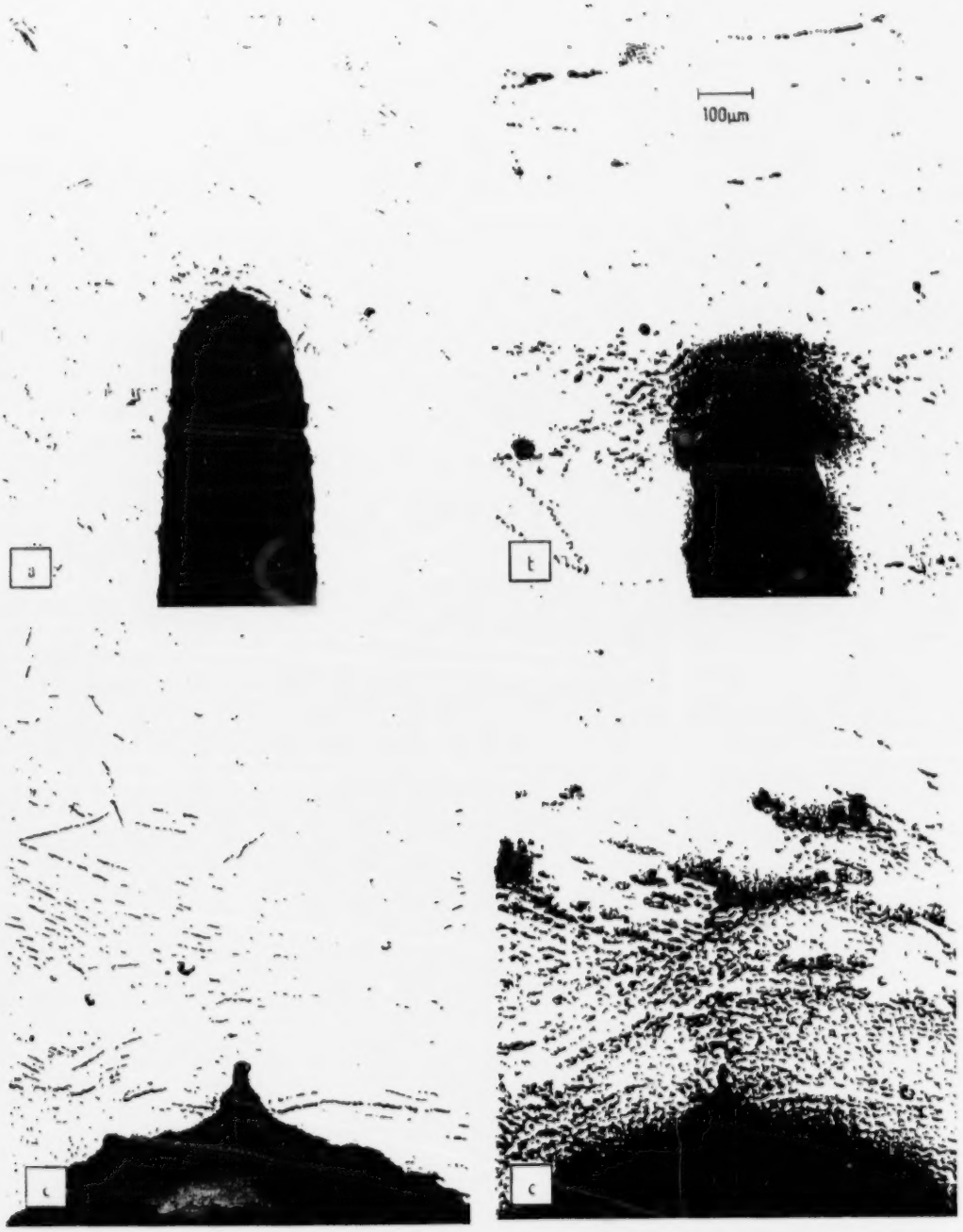

Figure 7. Sample #4, δ = 1.6 mm. (a. 100X electro-etched, b. 1000X electro-etched, c. 100X, ferro-fluid, d. 1000X, ferro-fluid).

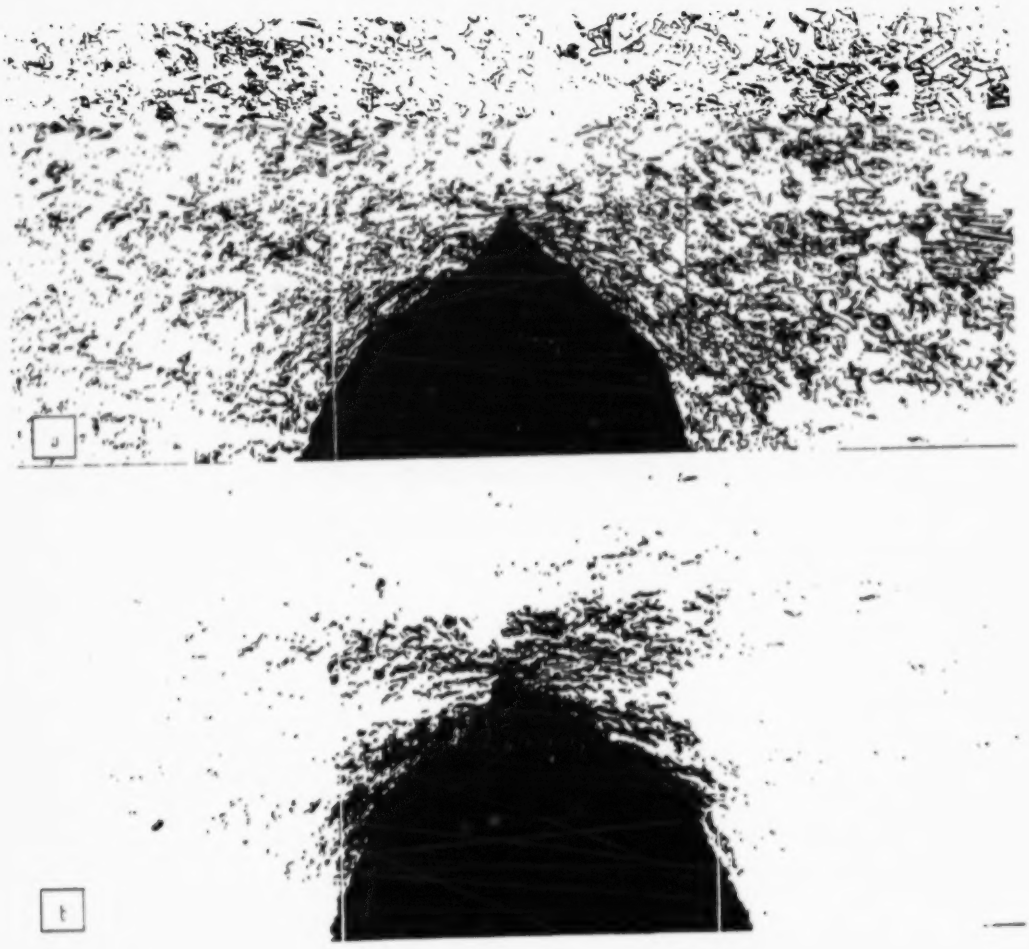

Figure 8. Sample #5, $\delta = 6$ mm, (a. 100X electro-etched, b. 100X, ferro-fluid).

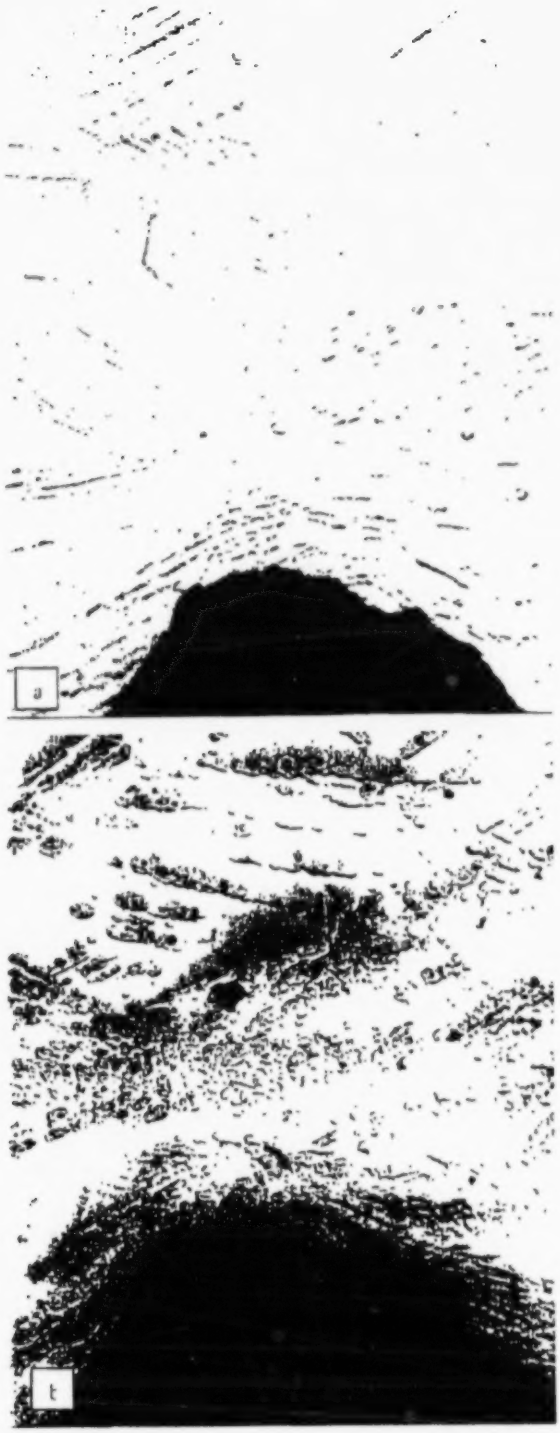

Figure 9. Sample #5, δ = 6 mm, (a. 1000X electro-etched, b. 1000X, ferro-fluid).

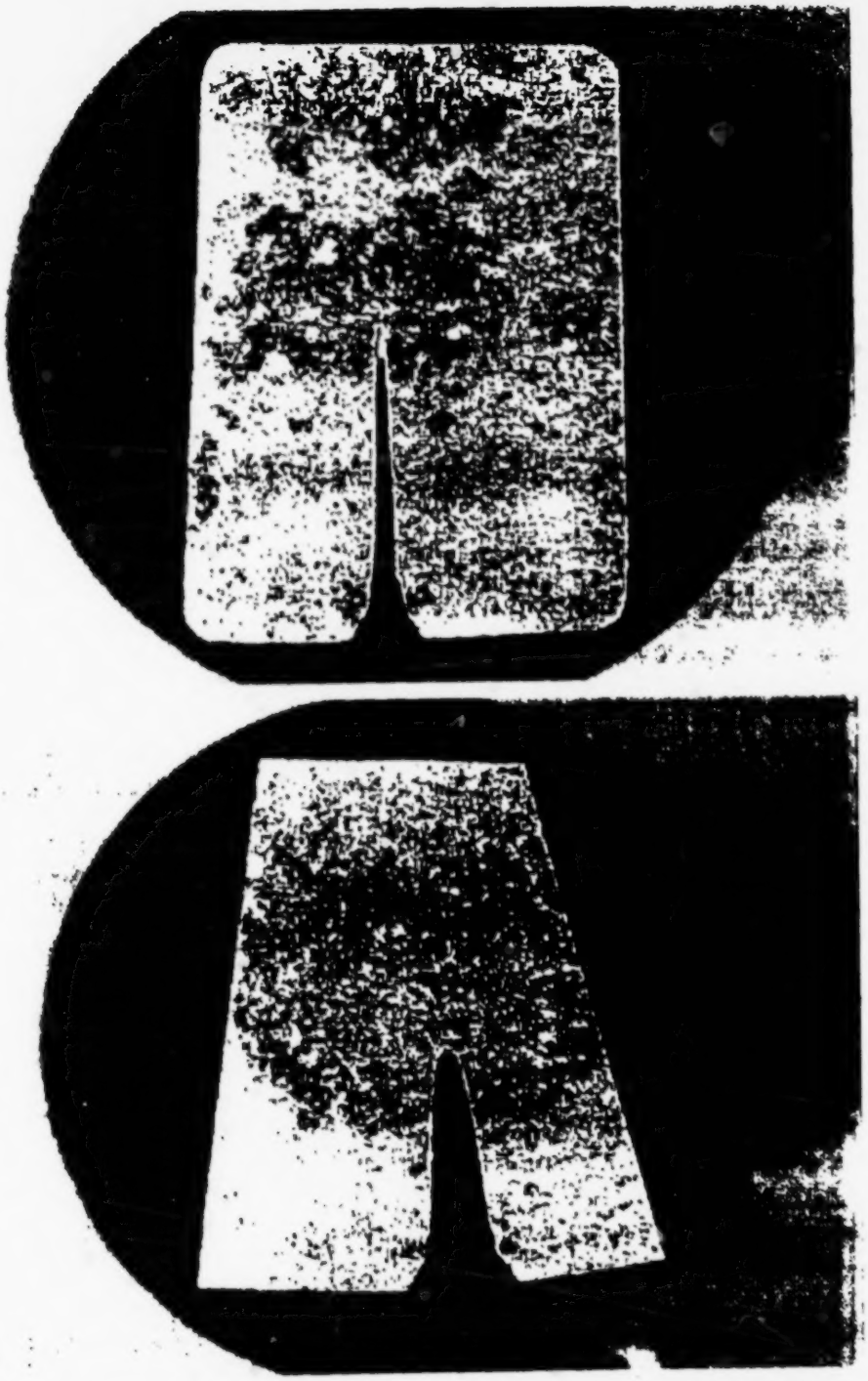

Figure 10. a. Sample #4 after recrystallization, b. Sample #5 after recrystallization.

DISCUSSION

Although it is possible to get a sense of the plastic zone using grain elongation it is unreliable for lower strains. In order for the grain elongation technique to offer the same insight as the ferro-fluid, a deflection of at least $\delta = 1.6$ mm had to be present.

As mentioned in the introduction, the characterization of the plastic zone by the ferro-fluid is dependent on the transformation from a metastable austenitic stainless steel to α-martensite through cold working. This approach is dependent on temperature, strain rate and composition. Localized heating during the precracking preparation of the samples and the final bending might be affecting the overall results. Also important are the strain rates. In this case they were kept low but a localized effect at the crack tip could affect the final outcome of the results. The consequences of the different composition of stainless steels with different carbon contents and how they would affect the resulting plastic zones are not known.

This procedure to reveal the plastic zone size is not strain level calibrated. It would be interesting to know what strain levels are indicated on the micrographs. Sample 1 for example had a plastic deformation radius of $r_y = 15$ µm, which is significant given that the crack was only 4 µm wide. The precracking performed on Sample 1 used a very low loading rate adhearing carefully to the precracking procedure keeping the load under 30 % of yield.

It is therefore possible that a relatively large plastic deformation radius is seen due to other causes. In the field of non-destructive testing, the magnetic particle inspection technique is used to assess surface cracks and defects. The break in conductivity is revealed by a change in the magnetic particle's pattern. With a non conductive epoxy filler in the crack it is reasonable to think that the same sort of effects can be found on Sample 1 due to the small plastic deformation radius.

By ploting the plastic deformation radius, r_y versus the stretch zone width (Figure 11) it is posible to establish a linear relationship between the two. The very first point on the graph has some plastic deformation but no stretch zone width due to the fact that all samples were started with the same 30% yield precracking.

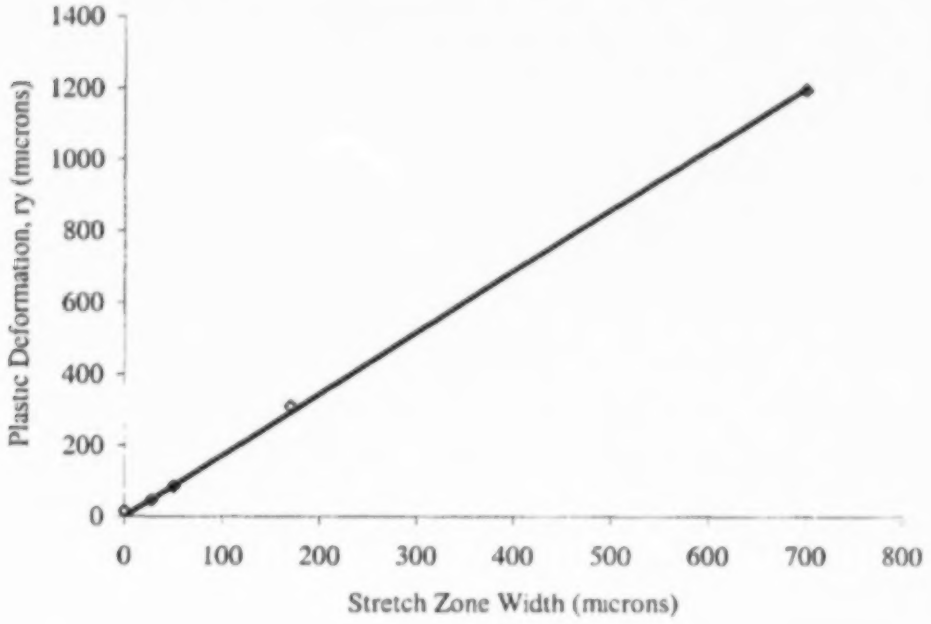

Figure 11. Plastic deformation (r_y) versus stretch zone width based on ferro-fluid information.

CONCLUSIONS

1. Ferro-fluid is a more reliable way to establish the plastic zone size and shape than visual observation using grain elongation. The temperature, the strain rate and the composition influence the results from this method and therefore the results will vary with different materials and experiment.

2. A linear relationship can be observed when comparing the plastic deformation radius (r_y) and the stretch zone width.

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